GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

PROJECT ADMINISTRATION DATA SHEET

[x] ORIGINAL [ ] REVISION NO. ______

Project No. G-35-616 GTRI/GIT DATE 7 / 24/ 84

Project Director: Dr. Doug Davis School/Lab Geo Sci.

Sponsor: National Science Foundation

Type Agreement: Grant No. ATM-8304897

Award Period: From 6/15/84 To 11/30/85 * (Performance) 2/28/86 (Reports)

Sponsor Amount:

	This Change	Total to Date
Estimated:	$	$ 160,000
Funded:	$	$ 160,000

Cost Sharing Amount: $ 9,302 Cost Sharing No: G-35-320

Title: "The Development of a Vacuum-Ultraviolet Photofragmentation/Laser Induced Fluorescence Sensor for Atmospheric Nh3 and Other Trace Gases"

ADMINISTRATIVE DATA

OCA Contact Lynn Boyd x4820

1) Sponsor Technical Contact:

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Defense Priority Rating: n/a

Military Security Classification: n/a

(or) Company/Industrial Proprietary: n/a

RESTRICTIONS

See Attached NSF Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval – Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of $500 or 125% of approved proposal budget category.

Equipment: Title vests with GIT

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COMMENTS:

*includes usual 6 month unfunded flexibility period.

Pre-award costs have been approved through OPAS. Also, advance project no, was assigned for $75,268.

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FORM OCA 4:383

GEORGIA INSTITUTE OF TECHNOLOGY OFFICE OF CONTRACT ADMINISTRATION

SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date 7-1-87

Project No. G-35-616 School/~~Lab~~ Geo. Sci.

Includes Subproject No.(s) N/A

Project Director(s) D.D. Davis GTRC / ~~GIT~~

Sponsor National Science Foundation

Title "The Development of a Vacuum-Ultraviolet Photofragmentation/Laser Induced Fluorescence Sensor for Atmospheric Nh3 and Other Trace Gases"

Effective Completion Date: 11/30/86 (Performance) 2/28/87 (Reports)

Grant/Contract Closeout Actions Remaining:

- [X] None
- [ ] Final Invoice or Final Fiscal Report
- [ ] Closing Documents
- [ ] Final Report of Inventions
- [ ] Govt. Property Inventory & Related Certificate
- [ ] Classified Material Certificate
- [ ] Other ____________

Continues Project No. ____________ Continued by Project No. ____________

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Other Duane H.
Angela DuBose
Russ Embry

FORM OCA 69.285

NATIONAL SCIENCE FOUNDATION
Washington, D.C. 20550

FINAL PROJECT REPORT
NSF FORM 98A

PLEASE READ INSTRUCTIONS ON REVERSE BEFORE COMPLETING

PART I—PROJECT IDENTIFICATION INFORMATION

1. Institution and Address	2. NSF Program	3. NSF Award Number
Georgia Institute of Technology School of Geophysical Sciences Atlanta, GA 30332	Atmospheric Sciences	ATM-8304897
	4. Award Period From 5/84 To 5/86	5. Cumulative Award Amount $270,200

6. Project Title "The Development of a Vacuum Ultra-Violet Photofragmentation/Laser-Induced Fluorescence Sensor for Atmospheric NH_3 and Other Trace Gases"

PART II—SUMMARY OF COMPLETED PROJECT *(FOR PUBLIC USE)*

The stated goal of this two year instrument development program was to demonstrate that a multi-photon laser-induced fluorescence technique, labelled by us as the "Vacuum Ultraviolet/Photofragmentation Laser-Induced Fluorescence" technique, could be successfully applied to the detection of trace atmospheric gases. The trace gas initially chosen for our feasibility study was ammonia, NH_3. After two years of effort, virtually all major obstacles involved in developing this new technology have been overcome. These include: (1) successfully reducing the 193 and 450 nm laser generated background fluorescence noise to workable levels (in the case of the 450 nm beam the noise has been reduced to zero); (2) convincingly demonstrating that in the 193 nm two-photon photofragmentation of NH_3, there is a significant yield of the metastable species, NH($b^1\Sigma^+$); (3) demonstrating the detection of the NH($b^1\Sigma^+$) species via excitation at 450 nm with preliminary calibration tests showing linearity over five orders of magnitude in NH_3 levels; (4) successfully demonstrating that potential chemical interferences, such as organic amines, were factors of 20 to 100 times lower in signal levels for comparable concentration levels of the amine compound and NH_3; (5) demonstrating that sampling line memory problems in detecting NH_3 could be overcome by using (a) passivated nickel as a construction material for sampliing lines and fluorescence cells and (b) using very high sampling flow rates (i.e. 125 to 150ℓ/min); and (6) demonstrating that the quenching of the metastable NH($b^1\Sigma^+$) state and the quenching of the fluorescing species NH($c^1\pi$) do not present major difficulties in the use of this new technique as applied to a broad range of tropospheric environments.

PART III—TECHNICAL INFORMATION *(FOR PROGRAM MANAGEMENT USES)*

1. ITEM *(Check appropriate blocks)*	NONE	ATTACHED	PREVIOUSLY FURNISHED	TO BE FURNISHED SEPARATELY TO PROGRAM: Check (✓)	Approx. Date
a. Abstracts of Theses	X				
b. Publication Citations				X(2)	Aug. 30, 1987
c. Data on Scientific Collaborators			X		
d. Information on Inventions	X				
e. Technical Description of Project and Results		X			
f. Other *(specify)*					

2. Principal Investigator/Project Director Name *(Typed)*	3. Principal Investigator/Project Director Signature	4. Date
Douglas D. Davis		5/87

NSF Form 98A (1-87) Supersedes All Previous Editions

Form Approved OMB No 3145-0058

INSTRUCTIONS FOR FINAL PROJECT REPORT
(NSF FORM 98A)

One copy of this report is due within 90 days after the expiration of the award. It should be submitted to the cognizant program officer in the NSF award letter at the following address:

National Science Foundation
ATTN: Program Officer
Division (Cognizant Division/Program)
1800 G Street, N.W.
Washington, D.C. 20550

INSTRUCTIONS FOR PART I

These identifying data items should be the same as on the award documents.

INSTRUCTIONS FOR PART II

The summary (about 200 words) must be self-contained and intelligible to a scientifically literate reader. Without restating the project title, it should begin with a topic sentence stating the project's major thesis. The summary should include, if pertinent to the project being described, the following items:

— The primary objectives and scope of the project.

— The techniques or approaches used (only to the degree necessary for comprehension).

— The findings and implications stated as concisely and informatively as possible.

NSF may disseminate the project report through the National Technical Information Service (NTIS) of the Department of Commerce. Authors should also be aware that the summary may be used to answer inquiries by nonscientists as to the nature and significance of the supported activity. Scientific jargon and abbreviations should be avoided.

INSTRUCTIONS FOR PART III

Items in Part III may, but need not, be submitted with this Final Project Report. Place a check mark in the appropriate block next to each item to indicate the status of your submission.

a. Self-explanatory.

b. For publications (published and planned) include title, journal or other reference, date, and authors. Provide two copies of any reprints as they become available.

c. Scientific Collaborators: provide a list of co-investigators, research assistants and others associated with the project. Include title or status, e.g. associate professor, graduate student, etc.

d. Briefly describe any inventions which resulted from the project and the status of pending patent applications, if any.

e. Provide a technical summary of the activities and results. The information supplied in proposals for further support, updated as necessary, may be used to fulfill this requirement.

f. Include any additional material, either specifically required in the award instrument (e.g. special technical reports or products such as films, books, studies) or which are considered to be useful to the Foundation.

Final Report

The Development of a Vacuum Ultra-Violet Photofragmentation/Laser-Induced Fluorescence Sensor for Atmospheric NH_3 and Other Trace Gases

Submitted To:

Atmospheric Sciences Division

National Science Foundation

Washington, DC 20550

Submitted By:

D.D. Davis

School of Geophysical Sciences

Georgia Institute of Technology

Atlanta, GA 30332

FINAL REPORT

A. Overview

Starting as early as 1975, the Georgia Tech Atmospheric Chemistry group began to explore the then emerging technique of single-photon laser-induced fluorescence (SP-LIF) as a means of detecting atmospheric trace gases under in-situ atmospheric sampling conditions. After many years of learning the pitfalls of adapting this laboratory analytical technique to atmospheric monitoring, we successfully developed systems for the detection of NO and SO_2 (see Bradshaw et al. 1982 a, b) and more recently for OH using the modified method 2λ-SP-LIF (Rodgers et al. 1982; Rodgers et al. 1984; Rodgers et al. 1985 and Davis et al. 1987). In spite of these successes, the limitations of both the simple SP-LIF and the more complex 2λ-SP-LIF techniques moved our focus in the direction of exploring the use of multi-photon LIF technology. It was our view that this new approach provided the means for eliminating (or greatly minimizing) both the problems of laser generated chemical interferences and the high levels of background fluorescence resulting from in-situ atmospheric aerosol species and/or organic films on the walls of the fluorescence chamber. Both problems can be resolved with multi-photon LIF techniques (especially as related to background fluorescence noise) because of the high probability that in a multi-photon approach fluorescence can be generated which is blue-shifted relative to the laser probe wavelength. This is in sharp contrast to the SP-LIF methodology where only red-shifted fluorescence can be produced as a detectable wavelength. As shown in the generalized schematic diagram in Fig. 1, the pumping wavelengths λ_1 and λ_2 are shifted to the red side of the sampled fluorescence λ_3. This permits the use of long wavelength blocking filters of very high optical density that can be used to remove both Rayleigh and Raman scatter from the λ_1 and λ_2 pump wavelengths as well as white background

PART IV - SUMMARY DATA ON PROJECT PERSONNEL

NSF Division Atmospheric Sciences

The data requested below will be used to develop a statistical profile on the personnel supported through NSF grants. The information on this part is solicited under the authority of the National Science Foundation Act of 1950, as amended. All information provided will be treated as confidential and will be safeguarded in accordance with the provisions of the Privacy Act of 1974. NSF requires that a single copy of this part be submitted with each Final Project Report (NSF Form 98A); however, submission of the requested information is not mandatory and is not a precondition of future awards. If you do not wish to submit this information, please check this box ☐

Please enter the numbers of individuals supported under this NSF grant.
Do not enter information for individuals working less than 40 hours in any calendar year.

*U.S. Citizens/ Permanent Visa	PI's/PD's		Post-doctorals		Graduate Students		Under-graduates		Precollege Teachers		Others	
	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.	Male	Fem.
American Indian or Alaskan Native												
Asian or Pacific Islander												
Black, Not of Hispanic Origin												
Hispanic												
White, Not of Hispanic Origin	3		1		1							
Total U.S. Citizens	3		1		1							
Non U.S. Citizens												
Total U.S. & Non-U.S. ..	3		1		1							
Number of individuals who have a handicap that limits a major life activity.												

*Use the category that best describes person's ethnic/racial status. (If more than one category applies, use the one category that most closely reflects the person's recognition in the community.)

AMERICAN INDIAN OR ALASKAN NATIVE: A person having origins in any of the original peoples of North America, and who maintains cultural identification through tribal affiliation or community recognition.

ASIAN OR PACIFIC ISLANDER: A person having origins in any of the original peoples of the Far East, Southeast Asia, the Indian subcontinent, or the Pacific Islands. This area includes, for example, China, India, Japan, Korea, the Philippine Islands and Samoa.

BLACK, NOT OF HISPANIC ORIGIN: A person having origins in any of the black racial groups of Africa.

HISPANIC: A person of Mexican, Puerto Rican, Cuban, Central or South American or other Spanish culture or origin, regardless of race.

WHITE, NOT OF HISPANIC ORIGIN: A person having origins in any of the original peoples of Europe, North Africa or the Middle East.

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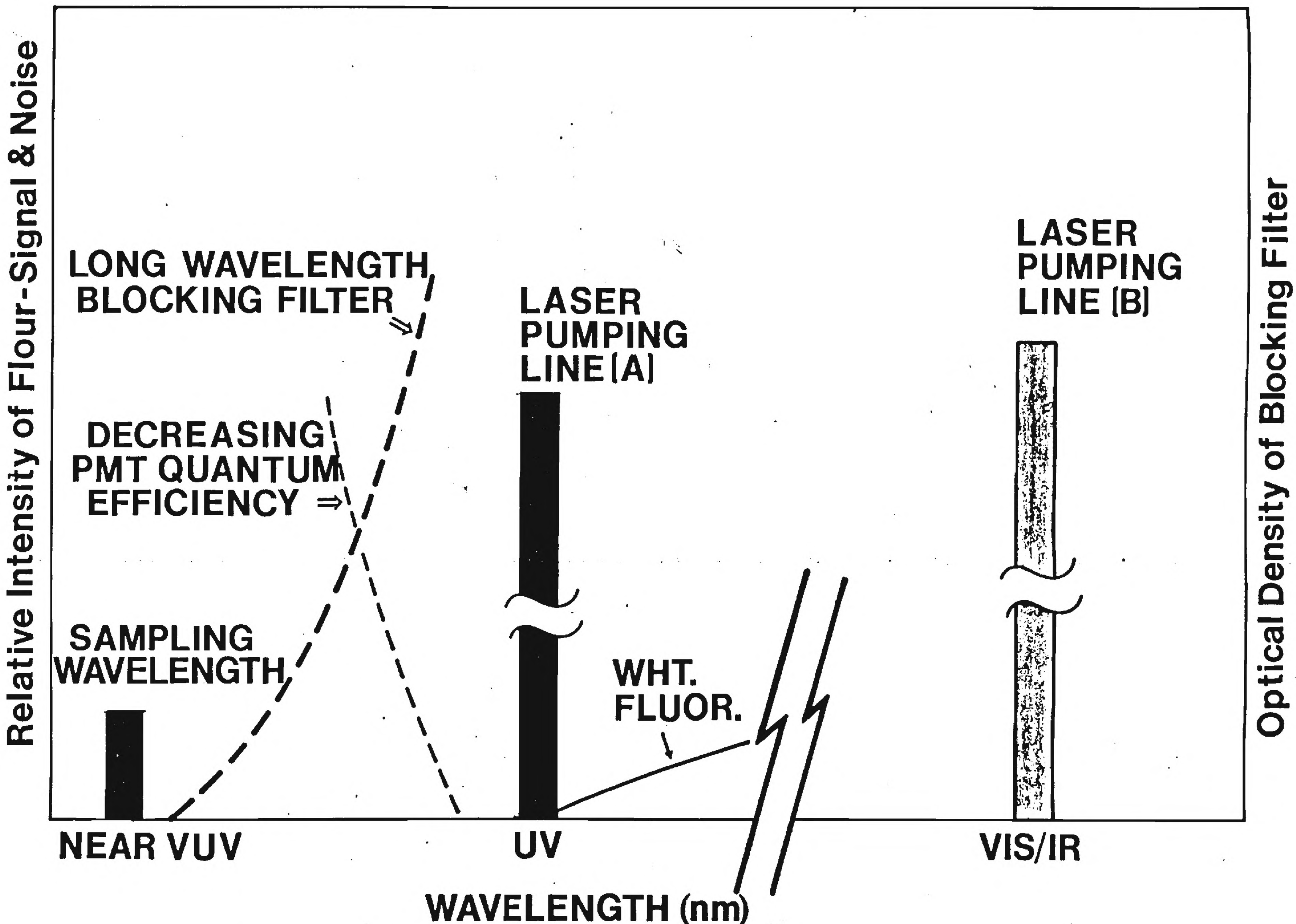
Figure 1

SAMPLING CHARACTERISTICS OF SEQUENTIAL TWO-PHOTON LIF SYSTEM



fluorescence noise from the λ_1 and λ_2 laser beams. With the further use of solar blind photomultiplier tubes (PMTs), one typically can reduce the total noise in a multi-photon LIF system to levels below the dark count of the PMT. Thus, with PMT time gating, multi-photon LIF systems become signal-limited rather than signal-to-noise limited as is characteristic of the SP-LIF methods. When operating in a signal-limited regime, the uncertainty in a given measurement is defined only by the uncertainty in the signal count. Thus, from photon statistics, one can estimate the fractional error in a measurement using the simple expression: $\sqrt{N_S}/N_S$, where N_S is the signal count for a pre-selected integration period. This suggests that multi-photon LIF techniques should inherently be very insensitive to changes in the background scattering environment (e.g. dust levels, fog, rain, ice crystals, etc.).

The first of the multi-photon LIF techniques examined in our lab was that illustrated in Fig. 1, i.e. the sequential two-photon LIF (STP-LIF) method. Initial efforts at developing laboratory systems of this type resulted in the detection of two chemical species, NO and Hg, (see Bradshaw and Davis, 1982; and Rodgers et al. 1982). Subsequent efforts by our group led to the development of a ground-based/airborne field system for the detection of NO (Bradshaw et al. 1985, Hoell et al. 1985, Hoell et al. 1987, Davis et al. 1987) which demonstrated in a convincing fashion that this new system indeed had all the basic characteristics suggested earlier in the text for a multi-photon LIF sensor under field sampling conditions. Additional species that have now been detected via the STP-LIF technique, under laboratory conditions, include OH and I_2.

Still a second form of multi-photon LIF detection of atmospheric trace gases is that proposed by Rodgers et al. in 1980. In this case, the approach involves photofragmentation LIF (PF-LIF). This methodology was directed at detecting molecules which either had very low fluorescence efficiencies under atmospheric conditions (e.g. NO_2) or species that had no bonding excited state and therefore could not be directly detected by a fluorescence method. The latter PF-LIF detection scenario has now been realized in field measurements of NO_2 and HNO_2 (e.g. Sandholm et al., 1987, Rodgers and Davis, 1987a,b and Davis et al., 1987).

Based on the success achieved with both the STP-LIF and the UV/PF-LIF systems, the Georgia Tech group proposed yet a third multi-photon detection scheme: Vacuum UV/Photofragmentation LIF (VUV/PF-LIF). As shown in Fig. 2 (a diagram for the NH_3 system), a key feature of this methodology is that photofragmentation is achieved in the near vacuum UV. Very important in this system is the fact that even at wavelengths as short as 193 mm, laser beams can be propagated one to two meters in air without major losses in energy. At the same time, one typically finds that numerous atmospheric trace gases, having small or insignificant cross sections in the UV, absorb very strongly (e.g. $\sigma \cong 10^{-17} cm^2$) in the near vacuum UV. However, a potential disadvantage of the VUV/PF-LIF approach is that unlike the UV/PF-LIF system, the condition of $\lambda_3 < \lambda_2$ and λ_1 is no longer true. Thus, the white background fluorescence noise from the λ_1 VUV laser must once again be dealt with. Unlike an SP-LIF system, however, another degree of freedom exists in the VUV/PF-LIF technique. If AB^* (Fig.3) is sufficiently long lived (as dictated by its natural lifetime, quenching by atmospheric gases or reaction with other species), one may time delay between the firing of the λ_2 and λ_1 lasers. By using appropriately adjusted delay times, the only remaining condition that must be met is that $\lambda_3 < \lambda_2$.

Upon examining the photodynamics of several molecules of potential atmospheric significance, two years ago we proposed that at least four species were likely to satisfy most of the conditions required of a VUV/PF-LIF system: NH_3, CS_2, SO_2, and H_2S. Of these, based on (1) the scientific need for reliable atmospheric measurements of NH_3, (2) what we believed to be reasonably reliable information on the photodynamics of NH_3, and (3) the availability of information on the physical and chemical characteristics of the photofragment $NHb^1\Sigma^+$, NH_3 was chosen as the first test molecule for our program.

The general detection scheme used in the case of NH_3 is that illustrated in Fig. 2, and given in summary form below:

$NH_3 + 2h\nu_1$ (193 nm) $\rightarrow$ NH $(b^1\Sigma^+)$

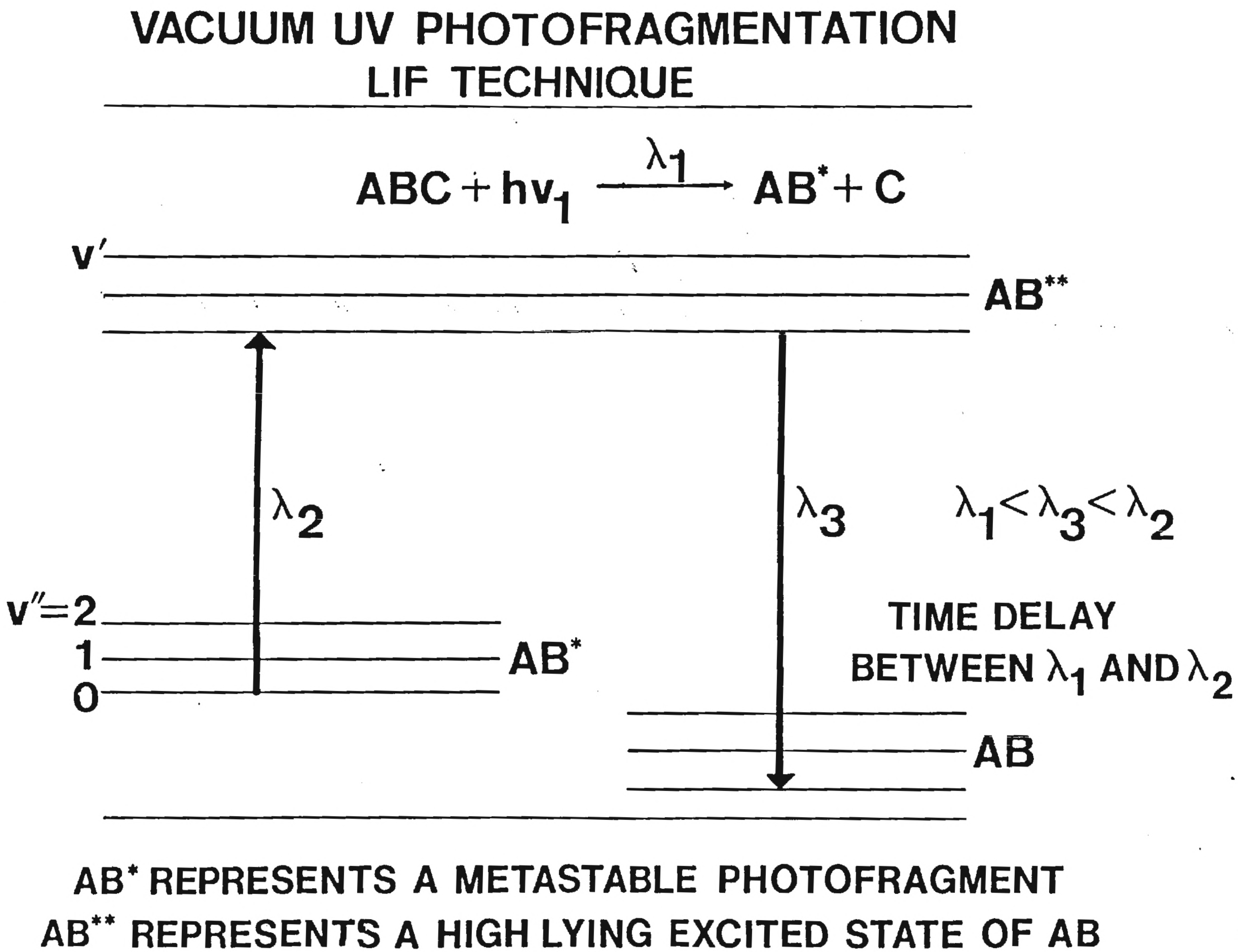
NH $(b^1\Sigma^+, v''= 0) + h\nu_2$ (450nm) $\rightarrow$ NH $(c^1\Pi, v'= 0)$

NH $(c^1\Pi, v'= 0) \rightarrow$ NH $(a^1\Delta, v = 0) + h\nu_3$ (326 nm)

B. VUV-PF/LIF Hardware Configurations

The evolution of the current VUV-PF/LIF ammonia detection system involved four distinct stages; however, only the final stage will be discussed here. The fourth and final instrumental configuration in the development of the VUV/PF-LIF NH_3 system is that shown in Fig. 3. The key elements of this system are: (1) an ArF excimer laser for generating the 193 nm photolysis beam; (2) a XeF excimer driven dye laser for generating tunable UV radiation in the spectral range of 450 nm; (3) an ambient fluorescence chamber from which 326 nm radiation from the $NH(c^1\pi)$ state is monitored; and (4) an NH_3 reference fluorescence cell used to normalize the signal level from the ambient chamber for changes in the 193 or 450 nm laser energies, or changes in beam overlape i.e. spatially and temporally. Fig. 4 shows a schematic diagram of the electronic set-up used in conjunction with the laser hardware.

Figure 2



VACUUM UV PHOTOFRAGMENTATION
LIF TECHNIQUE
$ABC + h\nu_1 \xrightarrow{\lambda_1} AB^* + C$
v′
AB^{**}
λ_2
λ_3
$\lambda_1 < \lambda_3 < \lambda_2$
v″=2
1
0
AB^*
TIME DELAY
BETWEEN λ_1 AND λ_2
AB
AB^* REPRESENTS A METASTABLE PHOTOFRAGMENT
AB^{**} REPRESENTS A HIGH LYING EXCITED STATE OF AB

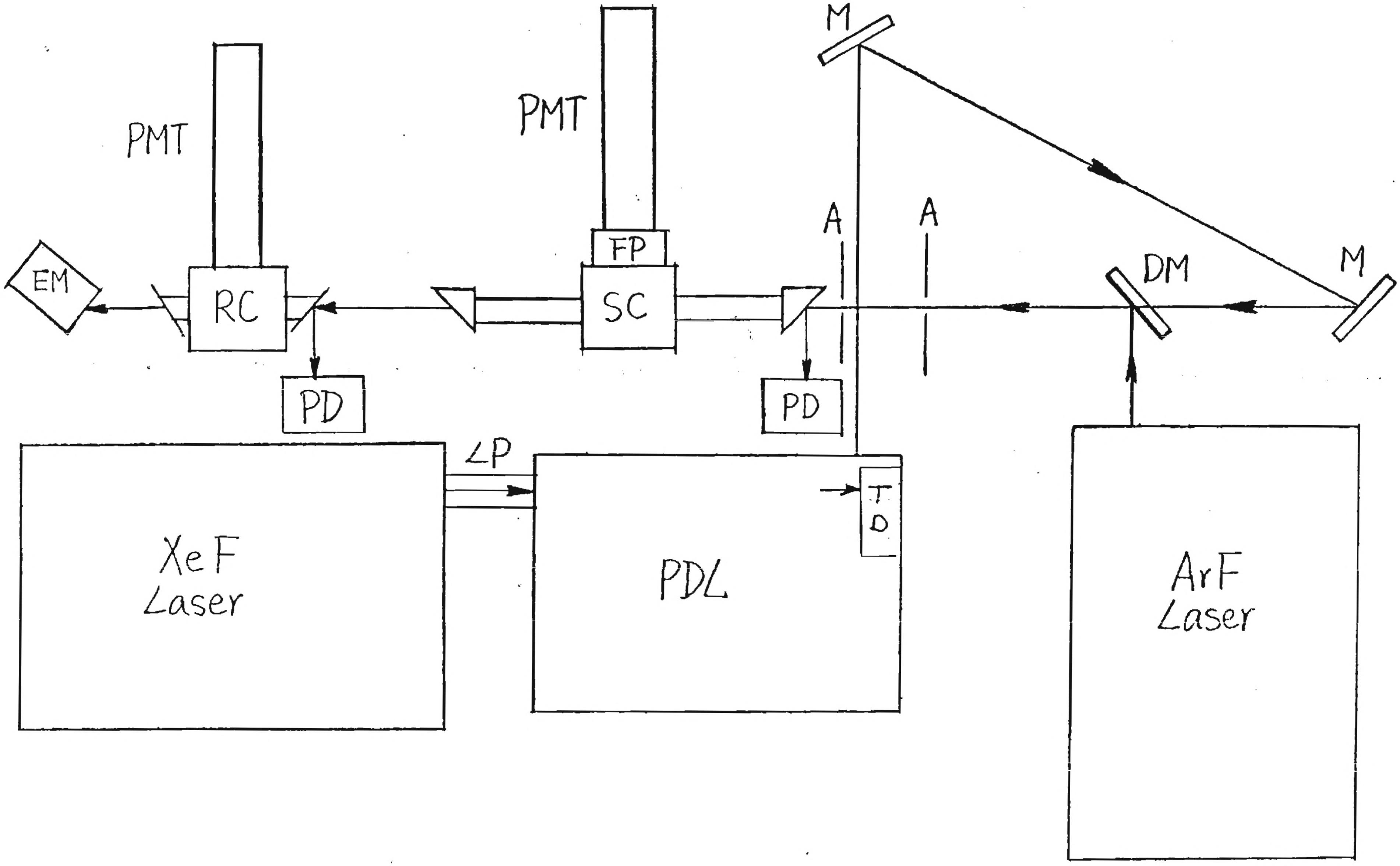


Fig. 3 - VUV/PF-LIF HARDWARE CONFIGURATION IV: CURRENT NH_3 DETECTION SYSTEM

(LP) Laser Pipe; (TD) Fast Timing Diode; (M) Mirror; (DM) Dichroic Mirror; (A) Aperture; (PD) Photodiode; (SC) Sample Cell; (FP) Filter Pack; (PMT) Photomultiplier Tube; (RC) Reference Chamber; (EM) Energy Monitor.

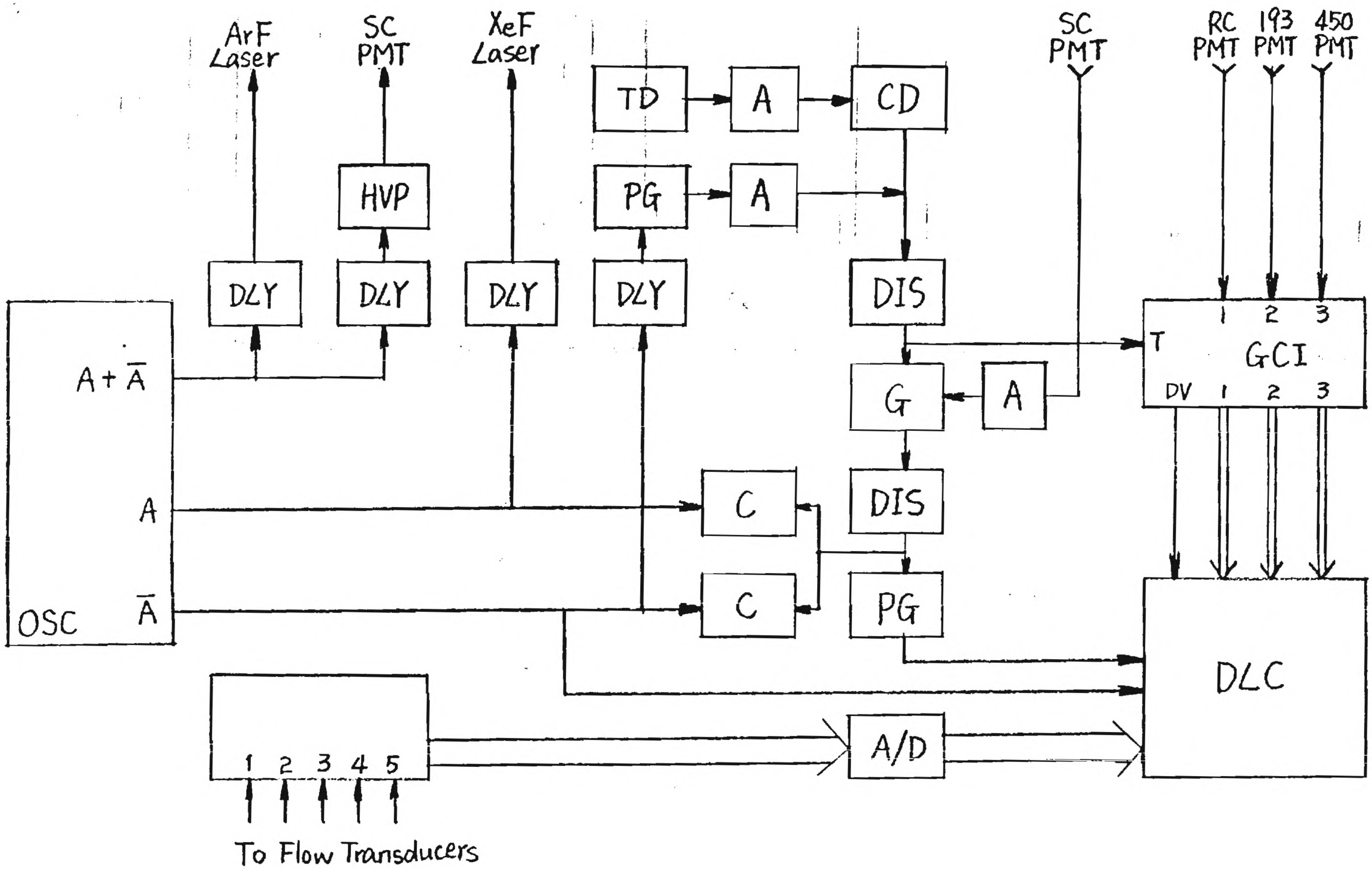


Fig. 4 - ELECTRONIC HARDWARE USED WITH LASER CONFIGURATION IV:

(OSC) Oscillator; (DLY) Electronic Time Delay; (HVP) High Voltage Pulser; (A/D) Analog-to-Digital Converter System; (PG) Electronic Pulse Generator; (TD) Trigger Diode; (A) Amplifier; (C) Counter; (G) Eletronic Time Gate; (SC) Sample Cell; (DIS) Discriminator; (GCI) Gated Charge Integrator; (DLC) Data Logging Microcomputer.

were likely to satisfy most of the conditions required of a VUV/PF-LIF system: NH_3, CS_2, SO_2, and H_2S. Of these, based on (1) the scientific need for reliable atmospheric measurements of NH_3, (2) what we believed to be reasonably reliable information on the photodynamics of NH_3, and (3) the availability of information on the physical and chemical characteristics of the photofragment $NHb^1\Sigma^+$, NH_3 was chosen as the first test molecule for our program.

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C. Summary of System Performance

After two years of effort, virtually all major obstacles involved in developing the VUV/PF-LIF NH_3 system have been overcome. These include (1) successfully reducing the 193 and 450 nm laser generated background fluorescence noise to workable levels (in the case of the 450 nm beam the noise has been reduced to non-measurable levels); (2) convincingly demonstrating that in the 193 nm two-photon photofragmentation of NH_3, there is a significant yield of the metastable species, $NH(b^1\Sigma^+)$; (3) demonstrating the detection of the $NH(b^1\Sigma^+)$ species, via excitation at 450 nm, with preliminary calibration tests showing linearity over five orders of magnitude in NH_3 levels; (4) successfully demonstrating that potential chemical interferences, such as organic amines, were factors of 20 to 100 times lower in signal levels for comparable concentration levels of the amine compound and NH_3; (5) demonstrating that sampling line memory problems in detecting NH_3 could be overcome by using passivated nickel as a construction material in conjunction with very high sampling flow rates (i.e. 125 to 150 ℓ/min); and (6) demonstrating that the quenching of the metastable $NH(b^1\Sigma)$ state and the quenching of the flourescing species ($NH(c^1\pi)$ do not present major difficulties in the use of this new technique as applied to a board range of tropospheric environments.

At this time, the detection limit of the VUV/PF-LIF system for NH_3 is 30 pptv (S/N=2) for a ten minute integration period. Alternatively, at 150 pptv (S/N=2), the integration time would be ~15 seconds. With the final optimization of the VUV/PF-LIF NH_3 system, a detection limit of 5 pptv per 5 min. integration period should be achievable.

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Rodgers, M.O. and D.D. Davis, "A UV Photofragmentation/Laser-Induced Fluorescence Sensor for the Atmospheric Detection of HNO_2", Applied Optics, (In Preparation).